FLODEX™ OPERATION MANUAL

TELEDYNE
HANSON RESEARCH

21-101-000-Rev. E—18 June 2019

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Document Revision History

REVISION	DATE	DESCRIPTION
A	11 Dec 1992	Initial release
B	16 July 1998	Added flow disks chart
C	25 Mar 2004	Added Section Three: Sales and Support, and Section Four: General Warranty.
D	09 Mar 2010	Updated bill of materials to correspond with figure 2 Stainless Cylinder Assembly (P/N 21-101-051).
E	18 June 2019	Updated to Teledyne format.

Table of Contents

Figures

Section 1 – Assembly Instructions

(See Figure 1 – Flodex final assembly

1. Insert the mounting post (2) into the base (1) while simultaneously inserting the shaft of the cylinder assembly (3) into the mounting post. Do not tighten the set screws.
2. Gently rotate the mounting post into the base until the cylinder assembly is approximately in the center of the base. Then, tighten the screws (9) in the base.
3. Lightly tighten the cap screw (6) holding the cylinder assembly shaft to keep the cylinder assembly centered in place while continuing assembly.
4. Push the funnel ring stand (5) over the mounting post and into the approximate position shown. Then loosen the cap screw (6), hold the cylinder assembly shaft, and slide the cylinder assembly in or out until the center of the cylinder exactly lines up with the bottom of the funnel. Tighten the screw holding the cylinder assembly shaft. Be sure the cylinder is vertically in line with the shaft (2) before tightening the screw.

(See Figure 2 – Stainless cylinder assembly)

5. Turn the release lever (9) until the lever arm drops. Insert a flow measurement disk (any size) with number side down by first removing the plastic ring retainer (3), inserting the disk and replacing the ring retainer with the disk in place.

6. Manually press the closure plate (5) against the disk and turn the release lever back to hold. Test by carefully and slowly moving the release lever forward until the closure plate falls without vibration into a vertical position.

   Note: The closure plate should gently fall into a vertical position without vibration. Change the tension on the screw (14) to adjust — “in” to stop sooner or “out” to stop later.

7. Move the funnel down until it is about 2 cm above the top of the cylinder. It is important that this dimension be maintained for a given test.

No.	P/N	Description
1	21-100-003	Base
2	21-100-004	Post
3	21-101-051	Cylinder assembly
4	21-101-013	Loading funnel
5	21-100-002	Funnel ring
6	41-100-010	Cap screw
7	21-101-019	Disk stand
8	21-101-055	Disk kit
9	¼-20 x ¾”	Mounting screw

Figure 1 – Flodex final assembly (p/n 21-101-050)

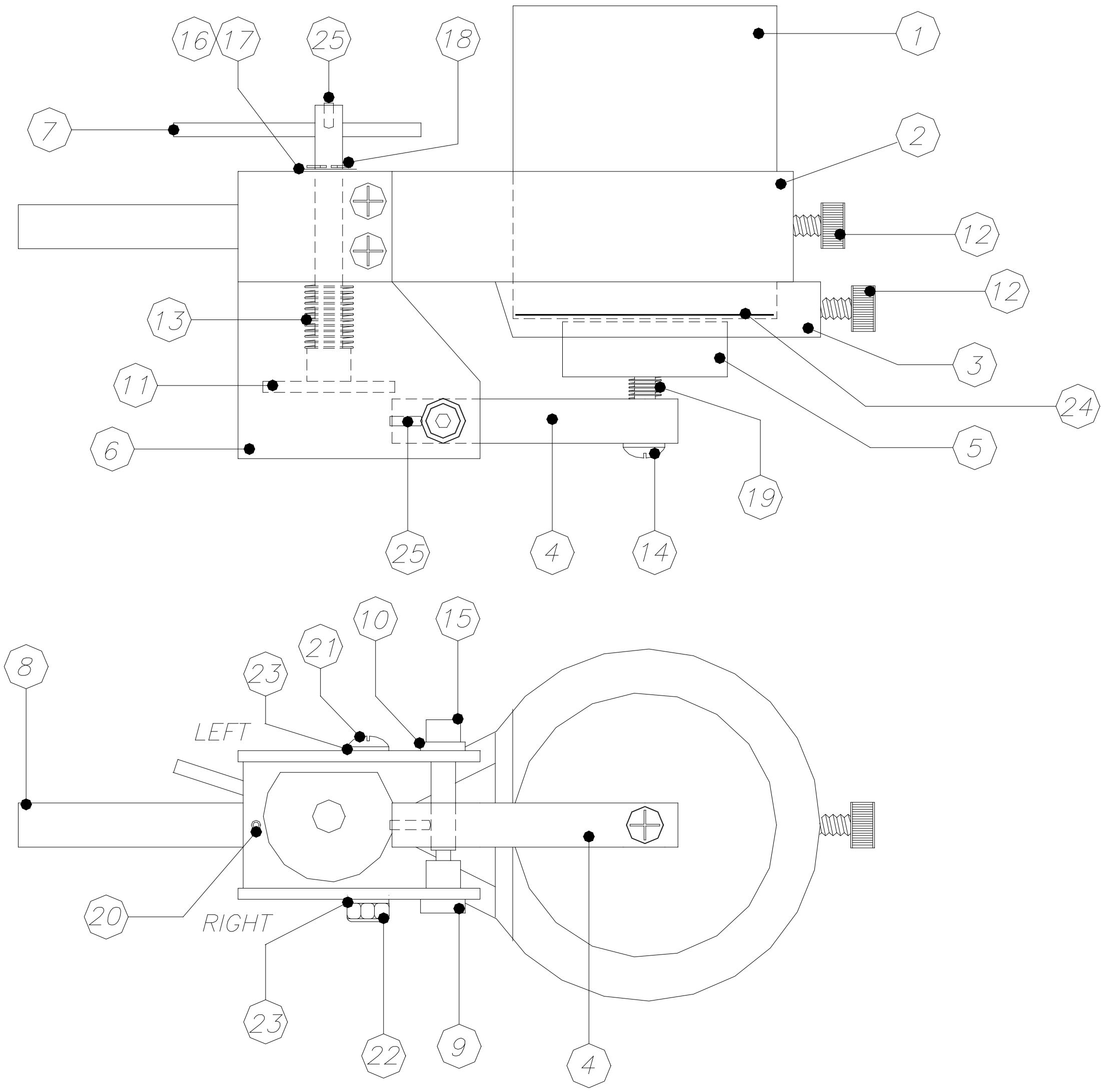

Figure 2 – Stainless cylinder assembly (p/n 21-101-051)

Note: See the following page for part numbers and descriptions.

This cylinder assembly is shipped completely assembled. It may be necessary to adjust dropping tension using adjusting screw (No. 14) for vibration-free drop after complete assembly.

Part numbers/descriptions for Figure 2 on previous page.

No.	P/N	Description
1	21-101-006	Stainless cylinder
2	21-101-004	Cylinder mounting clamp
3	21-101-010	Disk retaining ring
4	21-101-007	Release bar
5	21-101-022	Closure plate
6	21-101-002	Bearing plate
7	21-101-017	Release lever
8	21-101-012	Mounting rod
9	21-101-014	10-24 bushing
10	21-101-018	Bushing modification
11	21-101-052	Cam & shaft assembly
12	41-100-010	6-32 x 1/2 stainless screw & cap assembly
13	72-120-002	Tension spring
14	93-340-901	6-32 x 7/8 pan head screw, electropolish
15	91-418-001	10-24 x 1” with ¼” shoulder screw
16	91-413-018	Nylon washer, 0.01” thick
17	91-413-004	Nylon washer, 0.03” thick
18	91-406-005	Grip ring
19	91-445-050	Spring
20	92-540-205	10-32 x 1/4 stainless set screw
21	92-441-801	8-32 x 1.75” stainless pan head screw
22	92-440-013	8-32 stainless lock nut
23	92-400-011	#8 stainless lock washer
24	21-101-030	4 mm flow disc
25	92-340-205	6-32 x 1/4 set screw

Figure 3 – Inside hole diameters of flow disks

MM marking on disk	Inch; Tol. ± .003
4	.1575
5	.1969
6	.2362
7	.2756
8	.3150
9	.3543
10	.3937
12	.4724
14	.5512
16	.6299
18	.7087
20	.7874
22	.8661
24	.9449
26	1.0236
28	1.1023
30	1.1811
32	1.2598
34	1.3386

Section 2 – Operation

General

The Flodex employs technology developed by Dr. Alberto Gioia of Dow/Gruppo-LePetit, S.P.A. Milan, Italy, to determine a repeatable flowability index over an arbitrary scale of 4 - 40. This reliable index of powder flowability is used as a specification for raw materials utilized in formulating oral pharmaceutical dosage forms.

The Flodex presents a simple technique for the repeatable determination of power flow characteristics. This one simple test takes into account the numerous parameters that affect powder flowability such as particle size and shape, “fines”, unit surface, actual and bulk density, porosity, settling, and electrostatic charge. Because of its simplicity, the Flodex can be satisfactorily operated by non-technically trained personnel.

The Flodex determination of intrinsic flowability is based upon the ability of powder to fall freely through a hole in a disk. The flowability index is given as the millimeter diameter of the smallest hole the powder falls freely through in three successive tries.

Theory

A core of powder of the same diameter as a hole in the form of a cylinder surrounded by powder will fall through the hole if the weight of the core cylinder is greater than the friction of its side surface.

$$\pi r^2 hdg \geq 2\pi rhk$$

(weight of powder in core cylinder) ≥ (friction on side surface of core cylinder)

where:

h = height of core cylinder of powder

$\pi r^2 h$ = volume of core cylinder

g = acceleration of gravity (980 cm / sec^2)

d = non-tapped bulk density of powder

r = radius of hole (radius of core cylinder)

$2\pi rh$ = surface area of core cylinder of powder

k = coefficient of friction

By simplifying the previous equation, we can obtain:

Internal friction coefficient $k \leq 490rd$

where r is of sufficient magnitude to allow powder to fall freely.

The radius of a hole in the disk through which powder will fall freely is:

$$r \geq \frac{k}{490d}$$

In practice, when the weight of the core cylinder of powder overcomes the side internal friction, the core cylinder will move; if the powder has acceptable flowability, it will continue slowly until the plane is inclined enough to stop the flow.

Setup

1. The Flodex should be carefully assembled as directed in Section 1 – Assembly Instructions, steps 1 through 7. The funnel should be adjusted to meet the approximate center of the stainless cylinder assembly. It should be as close as convenient to the top of the stainless cylinder and no more than 2 cm above it.

2. Prepare to start a test with a 16 mm flow disk. A metal bowl or foil should be used to collect the sample. Metal and foil discharge electrostatic potential that builds up between particles of powder. For the same reason, the loading funnel is stainless steel.

   Note: Failure to repeat test data may result if the directions above are not followed. If the loading funnel is too high above the cylinder, the powder may not fill the load with the same untapped bulk density for each successive test. If the powder is not collected on a conductive sheet, it may acquire electrostatic charge from the previous test and if the same sample is re-run it may not pass through the same minimum hole.

Running the Test

A 50 g sample is standard, but enough powder should be used to fill the cylinder to within 1 cm or so from the top.

Loading

Load carefully, tapping the bottom of the funnel lightly so that the powder is introduced into the receptacle cylinder without packing. Do not tap the funnel enough to disturb the cylinder.

Note: Though the ring stand is resilient and dampens the vibration from tapping the funnel, it may be necessary to spoon-in small amounts of powder with poor flow characteristics as an alternative.

Operation

After loading, a minimum of 30 seconds should pass before starting the test to allow any possible formation of flocculi. As mentioned above, start with a 16 mm flow disk for powders not previously tested.

Slowly turn the release lever until the closure drops open without vibration. The test is positive when the open hole at the bottom is visible when looking down from the top. Do not tap or shake the Flodex instrument during this period of test time.

Evaluation

For positive results (hole visible), repeat with smaller and smaller disk holes until the test is negative. For negative results, increase the size of the disk hole until the test is positive.

The flowability index is the diameter of the smallest hole through which the sample will pass for three successive tests. Free-flowing powders will generally form an inverted cone with a consistent plane.

Section 3 – General Warranty

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Appendix A – Technical Bulletin on Intrinsic Flowability

See attached Teledyne Hanson Research Document 99-380-001 Rev. 06-19 “Flodex Intrinsic Flowability Technical Bulletin”

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Document 21-101-000 Rev. E

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FLODEX Technical Bulletin Reprinted from Pharmaceutical Technology, Feb. 1980

Intrinsic flowability: a new technology for powder-flowability classification

ALBERT0 GIOIA

THE FLOWABILITY of the powdered materials used in a tablet formulation is a major consideration in the production of this popular dosage form. ***Flowability*** may be defined as the powder's ability to flow evenly, by means of gravity and other forces, from the top to the bottom of the hopper and then on to the dosage, compaction, and crushing chambers.

In tableting applications, powder with a high degree of flowability offers several advantages. (1) A smooth downward flow of the material will minimize air-pocket formation. (2) The dosage chamber may be filled very accurately, which not only increases average weight and decreases variation in average-weight coefficient but also creates even pressure during compression, thereby lessening wear on machine parts. (3) Flowable powder increases the reproducibility of feed parameters, which results in consistent tablet hardness, friability, dissolution rates, and blood levels. (4) During compression, air is expelled well because of the powder's high degree of permeability, a quality which helps eliminate such tablet flaws as capping and splitting. (5) Finally, high production speeds may be maintained.

If flowability of the powder has been obtained by minimizing the percentage of fine powders, two additional advantages result: the limited surface area of the powder particles makes lubrication easy; and the reduced electrostatic and pneumatic dust sprays of fine powders help to increase yields and to keep rooms and machines clean.

Weight Uniformity

Attainment of weight uniformity is the prime objective in powder fractionation, but it is also something of a problem. First, although many pharmacopeias indicate the weight-variation limits acceptable in monodose forms, they do not delineate procedures for fractionating the bulk material in such a way that these limits are met. Moreover, the pharmaceutical industry's practice of using volumetric dosage in bulk fractionation ensures satisfactory uniformity only when the bulk material is liquid and nonthixotropic. For liquid materials, standard laboratory methodologies do exist for quantitative viscosity control under both virtually static and more or less dynamic conditions.

Powders to be fractionated should also have good flowability, but until now no laboratory methodology has been suitable for determining flowability indexes that were applicable to actual production. Augsburger and Shangraw perhaps came closest when, emphasizing that the main objective should be to attain a uniform weight, they experimented with some mixtures in tableting machines and considered the variation coefficient of the average weight as the flowability index.[1] Unfortunately, this evaluation system is flawed in that it is not absolute — it depends on working conditions and machinery used. Furthermore, the method requires that mixtures have reasonable flowability and lubrication. The average weight (CV) obtained in the production equipment, however, may be used as the point of comparison in laboratory tests, just as blood levels are used as points of comparison in dissolution tests.

Parameters Affecting Flowability

Many researchers have identified powder flowability with interparticle friction, which can be measured by various techniques — the flowability cone angle,[2] the tilting-table angle,[3] and the flow time under standard conditions,[4] to name a few. Gold et al. obtained a weight/time-variation layout under standard vibration conditions by sacrificing the clarity of data on a single variable.[5]

The data obtained by the aforementioned laboratory methods, however, do not represent a dependable estimate of the powder's behavior in the machine. I believe that these tests are unsuccessful because they are not directed at the true crux of the problem. In particular, it is incorrect to identify flowability with interparticle friction, as though powder particles were like glass or sand spheres.

The truth is that the parameters determining powder flowability are numerous — particle size, fines, unit surface, particle shape, actual density, bulk density, porosity, air permeability through the powder, electrostatic charge, humidity, settling effects, and cohesion forces (e.g., London and hydrogen) — and they have contrasting and interdependent influence. Flocculation alone — which is caused by cohesion forces, all other parameters permitting — can impair flowability, while very good flowability can be obtained with high-bulk-density powders consisting of almost spherical granules, without floccuii.

Flocculation and Its Origin

Flocculi are groups of low-cohesion fines; they cannot be isolated in a laboratory because they disintegrate easily in sieves. In production equipment. powder containing flocculi tends to adapt to the container (the hopper), a behavior similar to that of a liquid gelatinizing in its container.

Flocculation occurs when the large surface area — and therefore large contact area -- of fines favors cohesion forces. These are the same cohesion forces that greatly increase during compression to produce hard tablets; in powder bulk, however, these forces are too weak to force out the air created by the well-known embolic property of capillaries. Distances between granules therefore remain at 100 Å = 0.01 μm. Lactose, starches, and calcium carbonate provide classic examples of flocculation.

Humidity favors the occurrence of flocculation by increasing the contact surface among powder particles, by reducing electrostatic repulsion force since the dielectric constant of water is 80, and by favoring the leakage of electrostatic charge. On the other hand, a light electrostatic charge — obtained, for example, through sifting or by the addition of Cabosil — might contribute to the elimination of flocculi.

An Experimental Method

Principle behind the Method

The basis for this method is the powder's ability to fall freely through a hole in a plate. The diameter of the smallest hole through which the powder passes three times out of three is taken as the flowability index. This method has proved easily reproducible. Each trial is considered valid when the powder that falls involves the entire height of the powder (not to be less than 60mm).

Description of the Equipment

Very simply, the Dow-Lepetit device for testing intrinsic powder flowability (Hanson Research Corp., Northridge, California) consists of a cylinder with a series of replaceable disks — of different diameters — in the bottom; the hole is closed by a mobile shutter (Figure 1). The actual components of the system are as follows:

1. A stainless steel cylinder with an approximate capacity of 200 ml.
2. A series of stainless steel disks. Each disk has a precise hole in the center in graduated sizes differing l-2 ml in diameter and is easily attached to form a bottom for the cylinder.
3. A shutter that covers the hole and that may be quickly removed without vibration to allow the powder to flow through the selected hole.
4. An adjustable funnel for loading the sample cylinder with a free fall of the test powder.
5. A suitable container to collect the powder that flows through the unit.

The Procedure

The ring is secured to the bearing to allow the bottom of the funnel to be near *but not touching* the powder surface. A powder load of 50 g is then poured through the funnel into the middle of the cylinder. When loading is completed, 30 sec must be allowed for possible formation of individual flocculi or mass flocculation of the whole load (Figure 2).

Now the lever device is operated to open the hole in the disk quickly and without vibration. A very flowable powder will slowly flow through the small-diameter holes, leaving a cavity shaped like an upside-down, truncated cone (Figure 3). A powder that flocculates in bulk, on the other hand, will fall abruptly, forming a cylindrical cavity. If the experiment is negative — i.e., if the powder falls as just

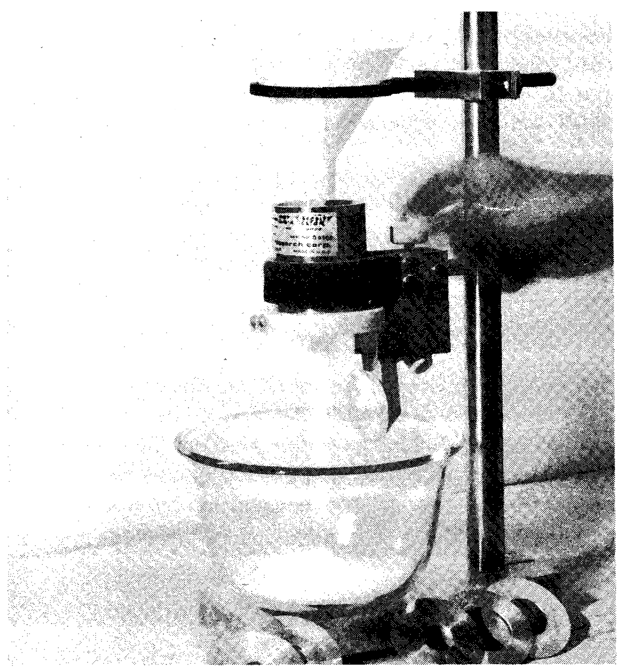

Figure 1: The equipment for testing powder flowability consists of a cylinder with a series of replaceable disks of different diameters — in the bottom; the hole is closed by a mobile shutter.

described — the powder must be tested again with a disk having a larger hole.

Physicomathematical Interpretation

It is simple to demonstrate the direct relation between the radius of the hole and the internal-friction coefficient -i.e., the viscosity — of the powder. Using K as the internal-friction coefficient, r as the radius in centimeters of the smallest hole that allows the powder to flow freely, and d as the nontapped bulk density of the powder in grams per milliliter, we can easily obtain

$$K \leqq 490 \cdot r\, d$$

Here, K is expressed in dynes per square centimeter, or poises; and 490 is equal to one-half the acceleration of gravity, or ½ g.

The weight of the cylinder of powder that is compelled to fall must be greater than the friction on the side surface of the cylinder itself:

$$\pi r^2 h \cdot d \,.\, g \geqq 2 \cdot \pi \cdot r \cdot h\, K$$

where

h = height of cylinder of powder
$\pi \cdot r^2 \cdot h$ = volume of cylinder of powder
g = 980 cm/sq sec (acceleration of gravity)
$2\pi \cdot r \cdot h$ = side surface area of powder cylinder
K = coefficient of friction per square centimeter

Simplified, the equation reads:

$$r \cdot d \cdot g \geqq 2 \cdot K$$
$$K \leqq \tfrac{1}{2} \cdot r \cdot d \cdot g$$
$$K \leqq 490 \cdot r \cdot d$$

It can also be said that a powder having viscosity K and nontapped bulk density d is sure to fall freely if

$$r \geqq \frac{K}{490 \cdot d}$$

For example, a powder with a density of 0.5 g/ml passing through a hole with a diameter not smaller than 24 mm (i.e., r = 1.2 cm) has a viscosity (or shearing strength) of K ≦ 294 poise = 29,400 cp.

The force initiating flow for powders having good flowability is the weight of the powder cylinder. Such powders flow from the top until the plane is inclined in such a way as to stop the flow.

Test Acceptability Limits

The limits that ensure good fractionation or granulation of the powder — that is to say, that ensure a low average weight — depend on machine type, working conditions, and powder composition. Our experience, however, allows us to make the following generalizations:

1. With Zanasi (Fratelli Zanasi SpA, Bologna, Italy) and MG 2 (MG2 Macchine Automatiche SpA, Bologna, Italy) capsulating machines, a good range is within disk-hole diameter limits of 10−24 mm.
2. With tableting machines, limits of the disk-hole size are 50%−120% of the diameter of the punch used.

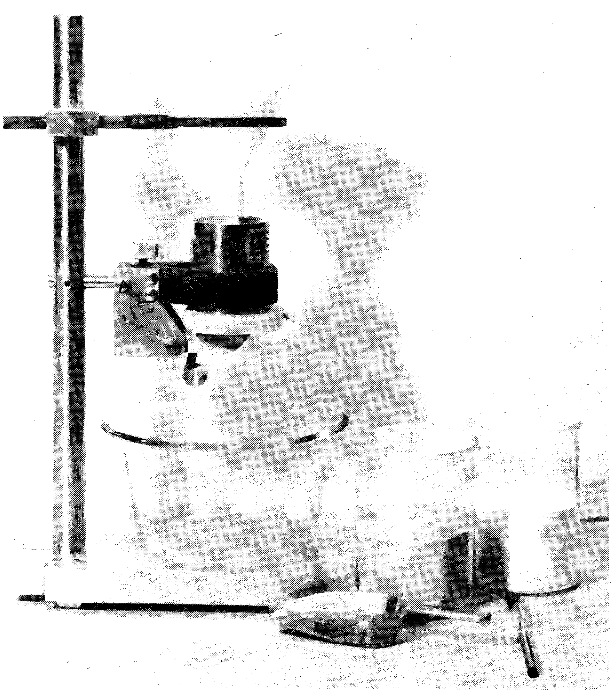

Figure 2: When loading is completed, 30 sec should be allowed for possible formation of individual flocculi or mass flocculation of the whole load.

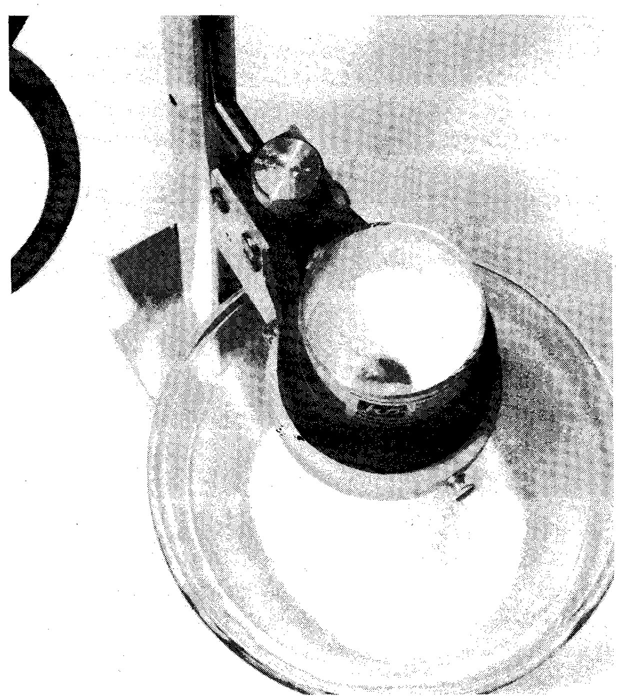

Figure 3: A very flowable powder will slowly flow through the small-diameter holes, leaving a cavity shaped like an upside-down, truncated cone.

Table I: A comparison of the flowability index and the coefficient of variation of typical pharmaceutical dosage forms.

Flowability Index (mm)	Coefficient of Variation of Average Weight*										
	Capsule Size 3600 Prod/Hr		Tablet Size								
			13,000 Prod/Hr				90,000 Prod/Hr				
	No. 1†	No. 0†	Dia. 6‡	Dia. 10‡	Dia. 13‡	Dia. 20‡	Dia. 8.5§	Dia. 11§	Dia. 11.55	17.5 X 7.15	19.5 X 8.55
4								1.09			
5						**0.93**					
6			1.10	1.17							
7				1.28					1.17		
8											1.32
10				1.32			0.60				
12					0.98						
20	0.52										
22	1.20						1.30				
24	1.76									0.87	
26	2.24	1.56									
30	3.33										
32										3.69	

* Capsules are calculated on the basis of contents without enclosure.
† Machine used: Zanasi-Lz-6 (2 punches).
‡ Machine used: Ronchi Rotary (8 punches).
§ Machine used: Manesty Express Rotary (20 punches).

The latter limits are much more restrictive than the former; this means that blends for tablets must frequently be granulated.

Conclusions

We have defined *powder jlowability* and outlined the parameters affecting its quality. Of particular importance are powder bulk density, which can be easily measured and can aid the powder's flow, and the presence of flocculi, which can obstruct powder flow.

In addition, we have defined the formulas for determining flowability. These formulas can be expressed as follows: flowability = l/diameter (in centimeters) determined by the test to be

$$\leq \frac{d \cdot g}{4 \cdot K}$$

The denominator is the viscosity coefficient and includes all the factors that oppose the powder flow. The numerator includes two parameters that aid the powder flow: bulk density and gravity acceleration. The electrostatic charge does not appear in this equation, although it may interfere indirectly. In fact, as mentioned earlier, a weak electrostatic charge can help eliminate flocculi, which reduces the K value. A strong electrostatic charge, you will recall, turns the fines away from each other, provoking a reduction in the nontapped bulk density of the powder and thus a reduction in flowability.

Low bulk density has two consequences: low weight and low flowability. During wet or dry granulation, therefore, both bulk density and flowability should be maximized. Moreover, prior to either capsule or tablet manufacturing, one should test both nontapped bulk density and flowability since these two parameters allow one to calculate the value of K.

In our laboratories at Gruppo Lepetit SpA (Milan, Italy), we have adopted flowability-index numbers that were obtained with the simple apparatus described herein as receiving and quality control specifications for all powders. The flowability index is now specified on all materials purchased from our suppliers.

Since these procedures were inaugurated, incidences of downtime and of failure of products to pass necessary tests (e.g., dissolution and product uniformity) have been reduced to zero. The value of establishing a limits of flowability index of feed powders to ensure product uniformity is suggested by the data in Table 1. The savings in production time and labor and the decrease in recalls have more than offset the investment made in adapting this uncomplicated test to purchasing, quality assurance, and manufacturing protocols.

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